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PATENT ABSTRACTS OF JAPAN

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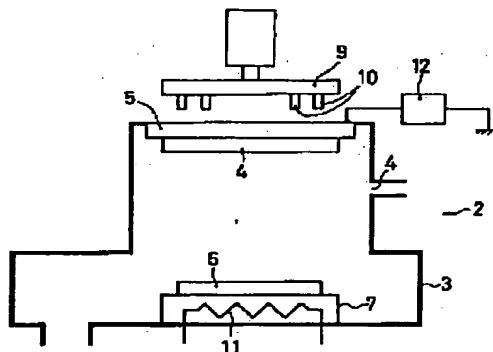
(54) FORMATION OF THIN TITANIUM NITRIDE FILM

(57) Abstract:

PURPOSE: To improve the film thickness distribution in a thin titanium nitride film while maintaining superior coverage.

CONSTITUTION: Gaseous argon is added to gaseous nitrogen in the range where, by flow rate ratio, $1/8 \sim 2$ (gaseous argon)/(gaseous nitrogen) $2 \sim 1/3$ is satisfied. Then, a titanium target 4 is sputtered while maintaining atmospheric pressure during film formation at a pressure of 21×10^{-1} Pa, by which a thin titanium nitride film is formed on a substrate 6. By this method, the thin titanium nitride film, improved in the filling up characteristics of fine pores and uniformity in film thickness distribution at the surface of the substrate, can be obtained.

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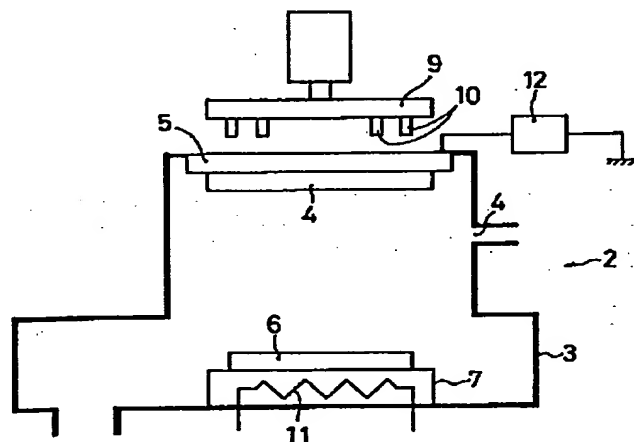
(54) 【発明の名称】 窒化チタン薄膜成膜方法

(57) 【要約】

【目的】 良好なカバレッジを維持したまま、窒化チタン薄膜の膜厚分布を改善する。

【構成】 前記窒素ガスに流量比で、
 $1/8 \leq \text{アルゴンガス} / \text{窒素ガス} \leq 1/3$
の範囲でアルゴンガスを添加し、成膜中の雰囲気圧力を
 $1 \times 10^{-3} \text{ Pa}$ 以下の圧力に保った状態でチタニウムターゲ
ットをスパッタして基板上に窒化チタン薄膜を成膜する
ことを特徴とする。

【効果】 微細孔の埋込特性がよく、基板表面の膜厚分
布が均一な窒化チタン薄膜が得られる。



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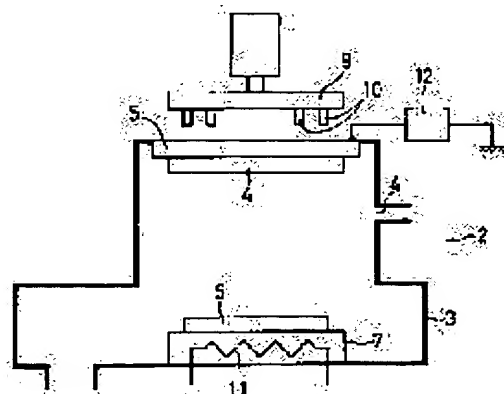
(21)Application number : 06-268873 (71)Applicant : ULVAC JAPAN LTD
(22)Date of filing : 01.11.1994 (72)Inventor : NAGATANI KOJI
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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the titanium-nitride thin film membrane formation method which embeds the micropore which started the membrane formation method of a titanium-nitride thin film, especially was prepared on the substrate by the manufacturing process of a semiconductor and electronic equipment by the titanium-nitride thin film.

[0002]

[Description of the Prior Art] By manufacturing processes, such as a semiconductor and electronic equipment, detailed-izing and multilayer-interconnection-izing of a design rule have progressed with high integration of a device in recent years. The technology which embeds conductive thin films, such as a metal thin film, to a contact hole, a through hole, etc. with a high aspect ratio is increasing significance with progress of such detailed-ized technology.

[0003] although the sputtering method and CVD are used from the former about this embedding technology, since there is much what has used gas detrimental to human being in CVD, an expensive gas facility and a damage elimination facility are needed, and there is a problem that the kind of thin film which can form membranes is also restricted, it is improving the sputtering method and the view of embedding the micropore of a high aspect ratio has become superior

[0004] By the way, the membrane formation by the sputtering method is the technology which introduce sputtering gas, impress negative voltage to the aforementioned target side under a predetermined pressure, produce electric discharge, carry out incidence of the ionized spatter gas molecule (ion) to a target, make the aforementioned substrate deposit the particle on the front face of a target begun to beat, and forms a thin film after carrying out opposite arrangement of the target in a vacuum tub, and the substrate and making it a vacua.

[0005] carrying out a deer, by the sputtering method of the conventional technology, as shown in drawing 4 (a), the particle 103 begun to beat from the target will carry out incidence to the micropore 102 prepared in the substrate 101 from various directions

[0006] Consequently, as shown in drawing 4 (b), particle 103' which has carried out incidence from across to a substrate will accumulate near opening of a micropore 102, an overhang 104 will be generated, and the shadowing effect which does not carry out ***** deposition will happen to the micropore pars basilaris ossis occipitalis 105. Consequently, there is a problem of becoming easy to generate an open circuit and a faulty connection near a micropore pars basilaris ossis occipitalis.

[0007] Although the spatter membrane formation method (the collimation spatter method) of having made it only the particle which carries out incidence perpendicularly to the micropore which has arranged the filter which prepared many slits between the target and the substrate to such a problem, and was prepared on the substrate reach a substrate is proposed, by such method, most particles which elutriated of the target adhere to a filter, and membrane formation efficiency falls. And the particle adhering to the filter is thin-film-ized, when this thin film separated, and it becomes dust and falls on a substrate, it has the problem that the yield falls, and it had not resulted in fundamental solution.

[0008] Then, the applicant proposed the so-called low voltage spatter membrane formation method which raises the rectilinear-propagation nature of a spatter particle by enlarging distance between a substrate and a target as compared with the case in the usual sputtering method, and keeping the ambient-pressure force under membrane formation at 1×10^{-1} or less Pa. According to this technology, the above-mentioned problem accompanying the pad of the micropore by the conventional sputtering method is solved, and, moreover, detailed stopgap on a substrate can be effectively carried out without problems, such as generating of dust.

[0009] However, although the embedded property in a micropore (coverage) was good when titanium (Ti) was used for the target, nitrogen gas was used as reactant gas and reactive sputtering was performed by the aforementioned low voltage spatter membrane formation method, in order to make a titanium-nitride (TiN) thin film form on a substrate, the thickness distribution of the titanium-nitride thin film on the substrate front face in which the micropore is not prepared did not become uniform. since the titanium-nitride thin film on this front face of a substrate is needlessness, although etching etc. needs to remove it unlike the titanium nitride inside a micropore, since the ununiformity of the thickness distribution on the front face of a substrate causes etching unevenness -- just -- dirty ** -- it was difficult, and when over etching was carried out, the titanium nitride inside a micropore also had un-arranging [much], such as *****ing

[0010]

[Problem(s) to be Solved by the Invention] It is in this invention offering the titanium-nitride thin film membrane formation method that the thickness distribution of the thin film on a substrate front face is uniform, maintaining [being created in order to improve un-arranging / of the above-mentioned conventional technology /, and] the good embedded property inside a micropore.

[0011]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, this invention carries out opposite arrangement of a substrate and the titanium target in vacuum atmosphere. In the titanium-nitride thin film membrane formation method which introduces nitrogen gas as reactant gas in the aforementioned vacuum atmosphere, carries out the spatter of the aforementioned titanium target, and forms the thin film of a titanium nitride to the aforementioned substrate to the aforementioned nitrogen gas by flow rate Argon gas is added in $1/8 \leq \text{argon gas} / \text{nitrogen gas} \leq 1/3$, and it is characterized by maintaining the atmosphere under membrane formation at the pressure of 1×10^{-1} or less Pa.

[0012]

[Function] a substrate and a titanium target -- ** -- if opposite arrangement is carried out, it sets in vacuum atmosphere, nitrogen gas is introduced as reactant gas in the aforementioned vacuum atmosphere and the spatter of the aforementioned titanium target is carried out, the thin film of a titanium nitride will be formed by the aforementioned substrate

[0013] The pressure in the case of this membrane formation is made into the pressure of 1×10^{-1} or less Pa, and if argon gas is added by flow rate to the aforementioned nitrogen gas in $1/8 \leq \text{argon gas} / \text{nitrogen gas} \leq 1/3$, the titanium-nitride thin film (TiN) which has a uniform thickness distribution, with a good embedded property maintained can be formed.

[0014] As for a thickness distribution, the flow rate of argon gas and nitrogen gas does not become good in less than $1/8$ value. Although the thickness distribution is uniform when argon gas is added on the other hand until it became a value exceeding one third, it turns out that it stops having shown the gold color peculiar to a titanium-nitride thin film, and became the middle-color tone (metal color which required yellow) of the insufficient shell of nitrogen, a titanium thin film, and a titanium-nitride thin film, and the $\text{Ti}_{1-x}\text{N}_x$ film with much titanium was formed. This $\text{Ti}_{1-x}\text{N}_x$ film has an inconvenient property for it being bad and using it as a barrier film.

[0015]

[Example] The example of this invention is explained based on a drawing.

[0016] With reference to drawing 1, 2 is the low voltage sputtering system used for this invention, and is equipped with the vacuum tub 3. This vacuum tub 3 is equipped with the gas inlet 4, the evacuation

mouth 3, and a target electrode 5 and the substrate electrode holder 7. The vacuum pump which is not illustrated is connected to the aforementioned evacuation mouth 3, the magnet plate 9 with a diameter of 320mm which has the magnet 10 arranged on a double concentric circle is formed in the rear face of the aforementioned target electrode 5, and the heater 11 is formed in the rear face of the aforementioned substrate electrode holder 7.

[0017] The aforementioned target electrode 5 is equipped with the titanium target 4 with a diameter of 300mm, and when it is constituted so that it may counter with the aforementioned titanium target 4 and wearing can do the substrate 6 with a diameter of 6 inches, and this substrate electrode holder 7 is equipped with a substrate, the aforementioned substrate electrode holder 7 is constituted so that the distance of this substrate and the aforementioned titanium target 4 may be set to 140mm.

[0018] Aspect ratio defined as the aforementioned substrate 6 by $A/R = a/e$ when the path of a and a base is set to e for the depth A/R The micropore of 2 is prepared. After starting the vacuum pump which does not carry out [aforementioned] illustration and changing the aforementioned vacuum tub 3 into a high-vacuum state, The aforementioned substrate 6 is heated at the aforementioned heater 11. from the aforementioned gas inlet 4 to the nitrogen gas of flow rate 26sccm The sputtering gas which added argon gas at a rate of flow rate 4sccm is introduced (the whole flow rate is 30sccm(s)). After waiting until it adjusted the exhaust speed and was stabilized in the value whose pressure is 5.7×10^{-2} Pa, when 10kW power was switched on by DC power supply 12 connected to the aforementioned target electrode 5 and sputtering was performed, the titanium-nitride thin film was formed by the aforementioned substrate 6.

[0019] The thickness of this titanium-nitride thin film was measured by nine points of the front face in which the micropore of the substrate 6 shown in drawing 2 is not prepared. When the position of the point P1 based on substrates is set to (0, 0), these nine points the eight remaining points It is expressed with P2 (35. 00 0), P3 (70. 00 0), P4 (00 - 35. 0), P5 (00 - 70. 0), and P6 (0 35.00), P7 (0 70.00), P8 (0 - 35.00) and P9 (0 -70.00) by setting the unit of a coordinate to mm.

[0020] The thickness of the above P1-P9 in one substrate is measured, and it asks for the maximum thickness and the minimum thickness, and is the following formula (thickness variation). When the thickness distribution was computed from $= \frac{\text{maximum thickness} - \text{minimum thickness}}{(\text{the maximum thickness} + \text{minimum thickness})}$, it was $\approx 6.8\%$. A thickness distribution has the good one where this value is smaller.

[0021] Moreover, the substrate which measured the aforementioned thickness distribution was cut and the titanium-nitride thin film which ground the cross section and was embedded at the aforementioned micropore 15 was observed. As shown in drawing 3, here The thickness d of the titanium-nitride thin film of the front face of the aforementioned substrate 6 The thickness c2 near the base center of the aforementioned micropore 15 and the thickness c1 and c2 near base ends are measured. The following formula (rate of average embedded), It was 37% when the rate of average embedded defined by $= \frac{c1}{d} + \frac{c2}{d} + \frac{c3}{d}$ was calculated from the measured value in three points of the points P1, P2, and P3 describing above.

[0022] Next, the titanium-nitride thin film was formed having used the membrane formation pressure as 6.5×10^{-2} to 2×10^{-2} Pa for the flow rate of nitrogen gas by having used the flow rate of 25sccm(s) and argon gas to 8sccm(s) preparing the micropore of an aspect ratio 2, equipping the aforementioned substrate electrode holder 7 with the substrate by which the titanium-nitride thin film is not formed, and keeping an interval with a titanium target at 140mm, thickness was measured similarly, and it asked for the thickness distribution and the rate of average embedded. As an example of comparison, argon gas was not added, but the thickness of the titanium-nitride thin film which carried out sputtering and formed membranes only with nitrogen gas was measured, and it asked for the thickness distribution and the rate of average embedded.

[0023] Moreover, the distance of a substrate and a titanium target was changed into 170mm and 200mm, the argon quantity of gas flow, the nitrogen gas flow rate, and the membrane formation pressure were changed further, and the aspect ratio formed the titanium-nitride thin film on another substrate which has the micropore of 2, and measured thickness. The thickness of the titanium-nitride thin film which

formed membranes only with nitrogen gas as an example of comparison was also measured.

[0024] The thickness distribution on the substrate front face of the titanium-nitride thin film which formed membranes under these membrane formation conditions and each conditions, and the rate of average embedded of a micropore are collectively indicated to Table 1.

[0025]

[Table 1]

成膜実験結果

* 投入電力 : 12 kW

基板／ターゲット 間距離	ガス流量 (sccm)		成膜圧力 (E-2Pa)	膜厚分布 (%)	平均の 埋め込み率 (%)	膜厚 (nm)	成膜速度 (nm/min)	備考
	アルゴン	窒素						
140	0	30	5.7	±12.3	35	425	81.7	比較例
	4	26	5.8	±6.8	37	384	74.0	
	8	25	6.5	±7.1	36	376	70.0	
170	0	30	5.7	±9.0	41	323	63.3	比較例
	4	26	5.8	±5.0	39	300	58.9	
200	0	30	5.7	±6.4	47	317	41.0	比較例
	3	24	4.9	±6.2	48	306	38.0	
	4	26	5.8	±4.9	47	292	36.0	
	6	26	7.2	±3.8	48	294	37.0	
	8	26	8.3	±3.4	48	294	37.0	

[0026] A thickness distribution is so small that the distance of a substrate and a titanium target is large as shown in the aforementioned table 1, and the rate of average embedded understands a bird clapper greatly.

[0027] However, even when this distance was enlarged, and its attention was paid to the distance between a substrate and a titanium target and it shortened [the membrane formation speed of a micropore pars basilaris ossis occipitalis fell and] on the other hand, according to the experiment, the membrane formation speed of a micropore pars basilaris ossis occipitalis did not necessarily become large. Therefore, when the membrane formation speed of a micropore pars basilaris ossis occipitalis is expressed by making distance into a parameter, a certain maximal value exists within the distance of the fixed range. This maximal value and the distance which gives the maximal value are influenced by an aspect ratio, the spatter pressure, etc.

[0028] However, it is not necessary to necessarily perform a spatter in the distance which gives the maximal value. In order for a membrane formation speed (36 nm/min) of the same grade as being based on the conventional sputtering method to obtain, it is checked by experiment that the distance between a substrate and a titanium target should just make it the range of about 100 to about 300mm.

[0029] In this case, the experimental result of Table 1 shows that it is desirable for the distance of a substrate and a target to make it 140mm or more.

[0030]

[Effect of the Invention] According to this invention, the embedded property in a micropore is good, and since a titanium-nitride thin film with the good thickness distribution on the front face of a substrate can be obtained, and the micropore which is a high aspect ratio is buried efficiently and put, the yield

improves.

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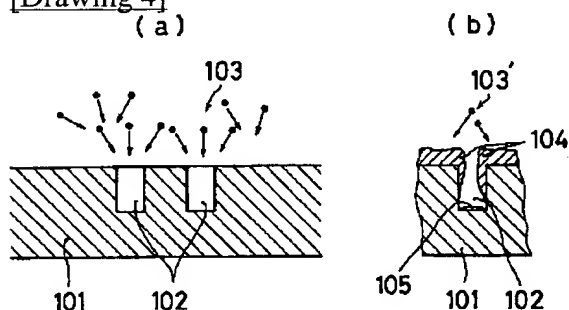
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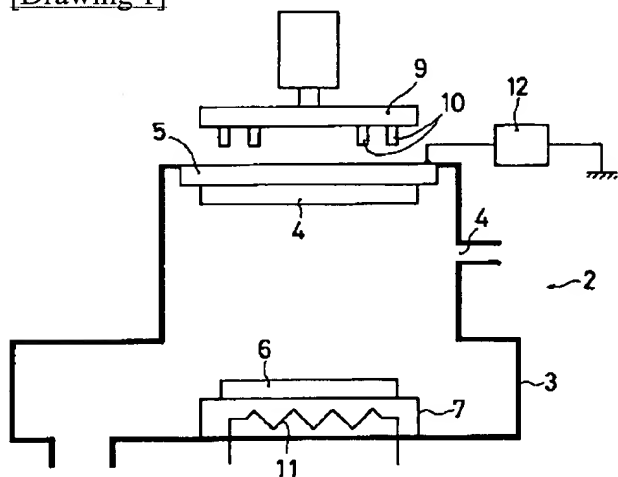
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DRAWINGS

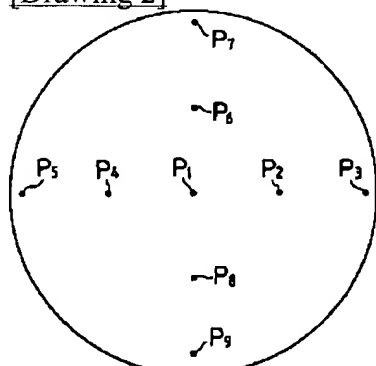
[Drawing 4]



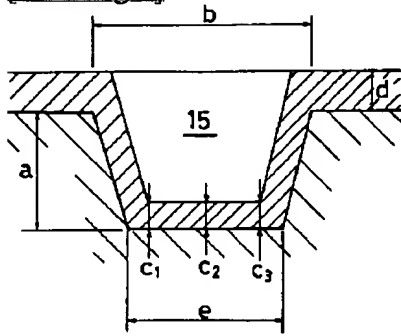
[Drawing 1]



[Drawing 2]



[Drawing 3]



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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] An example of the low voltage sputtering system used for this invention

[Drawing 2] Point of measurement of the thickness of a titanium-nitride thin film

[Drawing 3] The cross section of a micropore

[Drawing 4] (a) Drawing having shown the state where a thin film was formed by the conventional sputtering method Drawing showing the (b) shadowing effect

[Description of Notations]

4 Titanium target 6 .. Substrate

[Translation done.]